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ATOMIC WEAPONS RESEARCH ESTABLISHMENT

REPORT No. T 4/57

OPERATION BUFFALO

Target Response Tests

(Co-ordinator: E. R. Drake Seager)

The Decontamination of Radioactively Contaminated Drinking Water
in the Field

Maj. D. B. B. Janisch, R. A.

et al.

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July, 1957

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United Kingdom Atomic Energy Authority

ATOMIC WEAPONS RESEARCH ESTABLISHMENT

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OPERATION BUFFALO

The Decontamination of Radioactively Contaminated
Drinking Water in the Field

Major D. B. B. Janisch, R.A., et al

Summary

A method of removing dissolved fission products from potential drinking water supplies in the field has been developed at the Water Pollution Research Laboratory (DSIR). This report deals with field trials of a 1/20 scale apparatus that was used at Operation Buffalo. The apparatus worked effectively with the exception of removal of radioactive iodine. Recommendations regarding future development policy are made.

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1. Introduction

1.1 Historical

Late in 1954 it was suggested to the War Office that, with the introduction and use of tactical nuclear weapons, it would not always be possible for a unit in the field to have available supplies of drinking water that were not radioactively contaminated.

It was accordingly decided that an investigation should be made into possible methods of decontaminating water in the field. This work was carried out by the Water Pollution Research Laboratory (DSIR) at Stevenage, Hertfordshire, working on a brief prepared by the War Office Representative, the Army Medical Liaison Officer and the RAF Medical Representative at AERE, Harwell.

In this brief, it was suggested that the initial development of equipment should be centred around the 200 gallon 'Regimental Water Truck', leaving the larger continuously working equipments (the Paterson 3000 gal/h and the airborne 1000 gal/h) as a second stage development.

At the time at which the brief was prepared, medical opinion considered that the organ-seeking elements strontium and barium (bone-seeking) and iodine (thyroid-seeking), were the major hazard to humans drinking radioactively contaminated water. Since that date, opinions have changed and it is now considered that during the first few days after the burst, the dose to the gastro-intestinal tract from all the fission products, is of equal, if not greater, importance. It will be seen in the report that follows that although the method was developed around removing strontium, barium, and iodine, it effectively removes a large proportion of the other fission products as well.

As far as the Armed Services are concerned, the trials at present reported are the first to have been carried out under United Kingdom auspices.

The problem is not easily predictable as the actual elements, as well as their proportions, will vary depending on the circumstances of

the explosion (i.e., underground, ground or air burst, terrain over which exploded etc.), and the time after burst that the water is treated. It is therefore probable that the relative proportions of fission products, and their absolute concentration would differ with fall-out from (say) an underground burst in clay from those resulting from a low air-burst in a sandy area.

The present report records the work carried out at the WPRL and at Maralinga. The more complicated parts of the report relating to the detailed chemistry have been relegated to appendices for the sake of clarity. In addition, footnotes explaining certain chemical terms have been added for any readers who may not be familiar with chemical phraseology.

1.2 Outline of Work at WPRL

The Water Pollution Research Laboratory developed in the first place a laboratory apparatus designed to remove the organ-seeking elements strontium, barium and iodine in particular. This apparatus proved successful when used with solutions of salts of these elements and therefore as a second phase, an apparatus was developed with the capacity to deal with the relatively high flow-rates met with in field water purifying equipments.

Two forms of this apparatus were designed: one a full-scale equipment using an actual Metafilter from an Army 200 gallon water truck, and the other, a portable apparatus working on 1/20 scale. The former equipment, working as a laboratory mock-up and handling 200 gallons of water in 20 minutes, the flow-rate in the 200 gallon water truck, showed that the combined treatment would remove activity caused by iodine (^{131}I) as iodide, strontium (^{89}Sr and ^{90}Sr), barium (^{140}Ba), cerium (^{144}Ce) and zirconium/niobium ($\text{Zr}/^{95}\text{Nb}$).

The second of the equipments incorporated one Metafilter candle in place of the eleven used in the Service water vehicle; this equipment was taken on Operation Buffalo and operated there by the War Office Radiac User Trials Team*.

1.3 Principles Used for Removal of RA Elements

The early trials indicated that a cation-exchange resin was adequate for removing strontium and barium (because they behave chemically in a manner similar to calcium), and that silvered kieselguhr would remove iodine when present elementally or as iodide†.

Accordingly, the full-scale and 1/20 scale 'second stage' equipments used these materials.

* Major D. B. Janisch (War Office Representative, AERE), Major P. T. Thwaites and Captains M. H. Blakeney, T. T. Bunn, A. J. McCance and A. G. R. Makepeace.

† The element iodine may be present chemically as the element itself, or in an "acid radical" forming a salt with a metal. In these latter circumstances, the salt may be an iodide ($-\text{I}^+$), an iodate ($-\text{IO}_3^+$) or a periodate ($-\text{IO}_4^+$). The reaction with metallic silver will take place only when the iodine is present as the element or as an iodide.

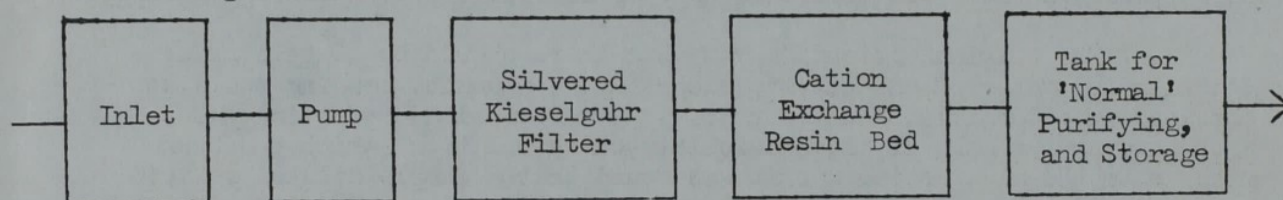
2. Task

The task was to produce an equipment that:

- (1) would remove radioactive strontium, barium and iodine and as much other activity as possible,
- (2) would do this at the relatively high flow-rates met in Service water purifying equipment,
- (3) would use relatively cheap materials, and hence, in its finished form, would not increase the cost of water purifying equipment unduly,
- (4) could be incorporated in existing water equipment.

3. Method

It was found by WPRL that the process indicated in the following flow diagram was the most suitable:



The ordinary Kieselguhr used in the Metafilter was replaced by silvered kieselguhr* ; the filter bed came immediately after the pump in order to have the filtration stage as soon as possible.

Following the Metafilter, a new bed was introduced, containing a cation exchange resin† between the filter and the storage tank. Associated with this bed were the necessary by-pass and regenerating fittings.

On the 1/20 scale equipment, which is the subject of this report, the complete Metafilter which is fitted to the 200 gallon water truck and contains eleven filter candles, was replaced by one candle, so that if ten gallons were treated in 20 minutes, the flow-rate through the filter would be $\frac{1}{20} \times 11 \times 100 = 55\%$ of that on the full-scale equipment. The size of the resin bed was similarly adjusted.

An annotated photograph of the equipment is shown in Figure 1. The method of setting it up is described in Appendix A.

* The normal variety is Metasil 'A'; the silvered variety was Metasil 'ALAG' manufactured by the Metafiltration Co. Ltd.

† ZeoKarb 225, manufactured by the Permutit Co. Ltd. This is a cation exchange resin in which ions of sodium are loosely held. Sodium ions will exchange with ions of calcium, strontium or barium when water containing the latter is passed through the bed. An ordinary domestic water softener employs an ion-exchange material.

3.1 Production of Contaminated Water

An attempt was made to collect fall-out from Round 1 (tower burst) and the top layer of a few square yards of the soil in the fall-out area was collected so that there was sufficient activity present in the sample. On sorting this soil, almost all of the activity was found to be concentrated in a small number of tiny glass-like spheres obviously consisting of fused sand. These were of course found to be almost insoluble in water; facilities were not easily available, neither was it considered expedient, to conduct the rather elaborate chemical processes necessary to bring this material into a neutral aqueous solution. Because of this relative insolubility, drinking water taken from this area would probably have been acceptable from the radioactive point of view*.

In order to obtain samples of contaminated water, the filter papers from one of the cloud-sampling aircraft in Round 3 (air burst), were used. These were macerated, and the soluble portion of the fission products leached out. The samples were collected shortly after F hour; the leaching out took place during D + 3, i.e., about 70 hours after burst, and the water was treated on D + 4, i.e., 92 to 100 hours after burst.

The original intention was that there should be 10 gallons of contaminated water of an activity such that an adequate reading would be obtained on a Contamination Meter No. 1 using the liquid counting tube. When however a sample of the original leaching solution (which had been made up to 450 ml) was tested, it was found that a sample diluted to 1:10 gave a reading of only 1.3 mr/h and one of 1:4 dilution, a reading of 4.0 mr/h. In other words, dilution of the original solution to only 1:10 would give a solution on the lower limit of contamination*. Further supplies of fall-out were not available and a decision had to be made regarding the procedure to be followed.

In view of the fact that a carrier was going to be added to the solution before treatment, it was decided that no harm would be done if the solution were diluted to the much larger volume required for the equipment, and an alternative means of measuring the activity used†.

The solution was accordingly diluted with distilled water to make up a total of 18 litres, i.e., a dilution of 1:40. All subsequent measurements of activity were made using an AERE Type 1221 Scaling Unit and a 20th Century Electronics liquid counter type M6H‡.

* i.e., by the accepted military emergency standard, of being fit for drinking up to $2\frac{1}{2}$ litres/man/day for 10 days.

† The carrier solution consists of (non-radioactive) chemically similar substances present in much larger quantities than the fission products. Hence any chemical or physical-chemical processes will affect the radioactive substances to a proportionately small degree. The solution used here gave concentrations of about 200 p.p.m. of calcium, 0.4 p.p.m. strontium and 0.4 p.p.m. barium.

‡ The M6H counting tube is similar in basic design to the liquid counter used with the Contamination Meter No. 1, for water contamination measurements. It consists of a thin-walled (beta-sensitive) Geiger counter with a re-entrant which enables a definite amount of the liquid to be measured, to be placed around the counter itself.

3.2 Decontaminating Procedure

The diluted "fall-out solution" was divided into four portions of approximately a gallon each. Each of these portions was passed through the equipment and treated. The circumstances of treatment varied slightly from portion to portion, e.g., some portions were passed through an ordinary (unsilvered) kieselguhr filter, while for others, the ion-exchange bed had been regenerated. Full details are given in Appendix B.

4. Results

Detailed results are given in Appendix C.

From these results, it will be seen that:-

- (1) Activity of untreated contaminated water fed into equipment (Portions A, B, C or D) *: 2460 counts/min.
- (2) Activity of 1st. gallon after treatment (A_t): 136 counts/min.
- (3) Activity of 2nd. gallon after treatment (B_t): 323 counts/min.
- (4) Activity of 3rd. gallon after treatment (C_t): 382 counts/min.

This indicates that approximately 95% of the gross activity was removed from the 1st. gallon during treatment but that the efficiency dropped to 85% on the subsequent two gallons. This was using a silvered kieselguhr filter and the same ion exchange bed throughout.

Treatment of portion D with an unsilvered kieselguhr filter and with an ion exchange bed that had already treated three gallons, removed only about 60% of the gross activity. However when this portion was reprocessed (as D_t) through a new ALAG filter and a regenerated ion exchange bed, virtually no activity was detectable in the effluent D_{tt} .

The effluent from the third gallon to be processed (C_t) was reprocessed through a filter bed and ion exchange bed that had already treated one gallon, and shewed a reduction of activity from 382 counts/min to 147 counts/min, a figure in line with the activity remaining in A_t .

When the ion exchange bed was regenerated, a sample from the washings shewed an activity of 3332 counts/min. It is to be assumed that the majority of the activity removed by the ion exchange bed was washed out in regeneration. Hence the activity from 18 litres of solution (portions A, B, C and D) that had been removed by the resin, was concentrated into the 1.25 litre of regenerant washings. Presumably therefore, had these washings been diluted to the original 18 litres, the activity recorded would have been

*The four portions of solution are referred to as A, B, C and D. After one pass through the apparatus, the effluents are referred to as A_t , B_t , etc. The effluents of portions treated twice are referred to as A_{tt} , etc.

$332 \times \frac{1.25}{18} = 232$ counts/min, as compared with 2460 counts/min recorded for the raw water, i.e., only 10% of the total activity. This however is of the same order as the proportion of strontium and barium likely to be present in the raw water.

Samples of both the raw water and of the third gallon after one and two treatments (C_t and C_{tt}) were returned by air to the WPRL who subsequently carried out radio-chemical analysis on them. Their results are given in Appendix D.

5. Conclusions and Discussion

The results in Appendix D, Table D1 show that the major active constituent in the effluent was iodine (only 31% of the total having been removed in the sample sent to the WPRL), while upwards of 91% of the strontium and barium was removed.

It should also be borne in mind that iodine occurs in fission products in a relatively high proportion.

The evidence in Appendix D, Section D2, suggests that a large proportion of the iodine activity in the air filter samples which WPRL had received, was present in a reducible anionic form such as iodate, and not, as has been suggested, as iodine or iodide.

If the proposed method of treatment is to be further developed, and if efficient removal of iodine continues to be specified, it will be necessary to find a method of treatment which will remove all the chemical forms in which radio-iodine is likely to occur in fall-out.

The equipment used at Maralinga was designed to remove strontium, barium and iodine (as elemental iodine or iodide) specifically.

Reference to Appendix D, Table D1 shows that these three elements constituted approximately 15% of the total activity in the raw sample, while in the effluents, the proportion of fission products other than iodine was fairly small. This shows that during the removal of Sr, Ba and I, the other fission products are removed to a considerable extent.

The reasons for the variations between the proportions of iodine removed after one pass (31% at Maralinga and 22% at the WPRL) and after a second pass (80% at Maralinga) are not clear. Some form of reduction of the iodine from a more highly oxidised form must presumably have taken place.

It will be noted that when the iodine in the solution used in the parallel series of WPRL trials was intentionally reduced chemically a similar proportion of the iodine was removed to that removed during the second pass at Maralinga.

5.1 Hazard to Operators

It must be remembered that whatever form the final equipment may take, nearly all the activity present in the raw water will be concentrated in the filter bed, ion-exchange bed and any other bed subsequently developed. When these beds are replaced or regenerated, as they will have to be after every 200 gallons on the Regimental Water Truck, or after equivalent times on other equipments, this activity is going to be washed out in a comparatively concentrated form.

If it is assumed that in its final form, the method is capable of removing 95% of the gross activity, then as a first approximation, there can be as much as 500 millicuries of active waste to dispose of after each 200 gallons (the calculation shewing this is given in Appendix E). Similarly with the Paterson 3000 gal/h equipment, over 5 curies per hour may have to be disposed of.

This is of no major consequence, but it should be borne in mind should development of this equipment for Service use proceed. For example, it will be necessary for the operator to keep at a distance from the beds except when it is actually necessary for him to approach, and for special precautions to be taken when removing the washings. As an example of the hazard, a dose-rate of the order of 1 r/h will be received at 2 ft from a point source of one curie of active material of similar energy to fission products.

The beta hazard will be of much less consequence as containers of sufficient thickness to be used on the water purifying equipment would adequately shield the operator against beta radiation. Similarly, if the washings were moved away for dumping in (say) jerricans, the material of the can plus shielding by the water of the solution, would adequately reduce the hazard to one of negligible proportions compared with the gamma hazard.

6. Recommendations

With the exception of iodine, the method developed by the WPRL shows considerable promise in removing fission products from potential drinking water supplies. A method of removing iodine in all its chemical forms still requires development: it is understood that this is feasible.

It is considered that sufficient basic research has been carried out on this method, to enable a Research and Development requirement to be laid down, should it still be considered operationally necessary to have an equipment of this nature.

In view of the fact that all the apparatus and trained personnel are at the WPRL, it is strongly recommended that any future work should be carried out there, if necessary on some form of extra-mural research contract.

7. Acknowledgment

The authors wish to express their gratitude to the Water Pollution Research Laboratory for their great assistance in this work.

APPENDIX A

Method of Setting-Up the Equipment

The ion-exchange column 'F' was charged with the resin, supplied in the fully regenerated condition, by placing it in water in the brine tank 'D' and washing it into the column through cock 'E'.

The filter bed 'B' was formed by removing the top of the filter and pouring in a slurry of the appropriate grade of kieselguhr, subsequently replacing the top and forming the filter bed on to the candle by pumping water through at a slow rate.

(The letters above refer to the annotated photograph in Figure 1.)

APPENDIX B

Decontaminating Procedure

(1) As mentioned in the main report, the 'fall-out solution' was diluted to a total volume of 18 litres, and was then divided into three volumes of approximately one gallon each and one of slightly less than one gallon. These are referred to in the remainder of the report as portions A, B, C and D. The same samples after treatment in the equipment are referred to as A_t , B_t , C_t and D_t . Any portions which were passed through the equipment twice are referred to as A_{tt} , B_{tt} etc.

(2) A background count was taken using the counting tube filled with distilled water. Following this, a count was made of a sample of the original solution diluted 1:100. An approximate cross reference between the readings obtained with the Contamination Meter No. 1, and the AERE 1221, is therefore possible.

(3) After washing out the counting tube, the background was taken again, and then a sample of the 1:40 solution was counted (i.e., a sample from A, B, C or D).

(4) Portion A was then passed through the equipment and the effluent run off into a 1 gallon glass jar (as sample A_t).

(5) Portion B was then passed through the equipment immediately after A and was run off into a separate jar as B_t .

(6) Portion C was then treated similarly.

(7) Concurrently with this, counts were made on samples from B_t , C_t and A_t (in that order).

(8) After passing portion C through the equipment, the filter bed was washed out and the Metasil ALAG (silvered kieselguhr) was replaced with Metasil A (ordinary kieselguhr). The ion exchange bed was not touched. The remaining $\frac{3}{4}$ gallon (portion D) was passed through this new filter and a sample from the effluent D_t was counted.

(9) The ion exchange bed was then regenerated with brine solution and a sample from the washings was counted. The total volume of brine + washings was 1250 ml.

(10) The Metasil A in the filter was then replaced by fresh Metasil ALAG, and B_t was again passed through the equipment which now had a new filter and a regenerated resin bed. A count was made on a sample from the effluent D_{tt} .

(11) C_t was also reprocessed and a count made on a sample from effluent C_{tt} .

APPENDIX C

	Relevant Section in Appendix B	Time of Count (from F hour)	Period of count, min	Count	Count, counts/min	Corrected Count, counts/min	Corrected Count Less b/g	Remarks
a. Background count at start using distilled water	(2)	91 h 35 m	5	72	14	14	-	
b. Sample of original solution diluted 1:10	Report Section 3.	91 h 40 m	-	-	-	-	-	1.3 m ² /h on Contamination Meter No. 1.
c. Sample of original solution diluted 1:3	Report Section 3.	91 h 45 m	-	-	-	-	-	4.0 m ² /h on Contamination Meter No. 1.
d. Sample of original solution diluted 1:100	(2)	92 h 0 m	5	5135	1027	1034	1020	
e. Sample of original solution diluted 1:40	(3)	92 h 10 m	5	12195	2439	2479	2465	
f. i.e., portions A to D	(3)	92 h 15 m	5	12129	2426	2466	2452	
g. B/g count after (f)	-	92 h 25 m	5	805	161	161	-	
h. Sample from effluent B _t	(7)	93 h 15 m to 94 h 45 m	90	43454	483	484	323	
j. B/g count after (h)	-	94 h 55 m	5	620	123	123	-	
k. Sample from effluent C _t	(7)	95 h 15 m	5	2518	504	505	382	
l. Sample from effluent A _t	(7)	95 h 20 m	5	1287	259	259	136	
m. Sample from effluent D _t	(7)	95 h 40 m	5	5379	1076	1083	960	
n. B/g count after (m)	-	95 h 45 m	5	680	135	135	-	
p. Sample of regenerant washings	(9)	96 h 25 m	5	16950	3390	3467	3332	
q. Sample from effluent D _{tt}	(10)	96 h 45 m	5	694	139	139	4	
r. Sample from effluent C _{tt}	(11)	97 h 10 m	5	1411	282	282	147	

APPENDIX D

Results of Laboratory Trials at WPERL

D.1 Examination of Samples of Water Returned from Maralinga

These were samples of untreated water (i.e., A, B, C or D), and of C_t and C_{tt} .

TABLE D1

Sample	Hardness as CaCO_3 , P.P.M.	μc per litre at D + 13			
		Gross $\beta \gamma$	^{131}I	^{89}Sr	^{140}Ba
Untreated (Sample C)	196	0.887	0.107	0.0177	0.052
3rd. gallon after one treatment (C_t)	1.9	0.087 (90%)	0.074 (31%)	0.00025 (99%)	0.0046 (91%)
3rd. gallon after two treatments (C_{tt})	2.5	0.030 (97%)	0.0217 (80%)	<0.00001 (> 99%)	0.000113 (99.8%)
<u>NOTE:</u> Percentages in brackets indicate percentage removal represented by the entry immediately above.					

D.2 Parallel Laboratory Trials

For a parallel but separate series of laboratory experiments, a contaminated water was made up by adding 50 ml of the extract from a sample of an air filter from Round 1 to 20 gallons of unfiltered reservoir water from the Lea Bridge Works of the Metropolitan Water Board. WPERL then used an apparatus developed by them for investigations into the removal of iodine by silvered kieselguhr.

The water was pumped through the Metafilter charged with 50 grams of Metasil ALAG at a rate of 13.8 gallons in 20 minutes. This corresponds to about 60% of the maximum rate employed in the 200-gallon unit. Part of the filtrate was passed through a small cation exchange column at a rate corresponding to the use of an 8-litre resin column at the rate of 200 gallons in 20 minutes. The regeneration level was equivalent to 20 lb per ft^3 .

Analysis of representative samples of the water before and after treatment gave the results show in Table D2.

TABLE D2

Results of Laboratory-Scale Decontamination of Water

Sample	μc per litre at D + 7			
	Gross $\beta \gamma$	^{131}I	^{89}Sr	^{140}Ba
Influent	0.274	0.032	0.013	0.0249
Effluent	0.112	0.025	< 0.0012	0.00025
Percentage removal	59	22	> 91	99

In view of the low proportion of iodine activity which was removed by this treatment, an additional experiment was carried out using active material from a Round 3 air filter. A 20-gallon batch of contaminated water was prepared as in the preceding experiment and was passed at the rate of 1 gallon per minute through the Metafilter charged with Metasil ALAG. Analysis of samples of water taken before and after filtration showed no detectable change in the content of active iodine.

D.3 Notes of the Chemical Form of Iodine in Water Contaminated by Fall-Out

- (1) A 10-ml aliquot of the filter extract used in the preceding experiment was treated by addition of 1 mg iodate carrier, 1 drop of 4N hydrochloric acid, and a slight excess of sulphurous acid solution. The solution was neutralised, diluted to 5 litres with unchlorinated water, and passed through a Metafilter charged with Metasil ALAG at a rate of 1 gallon per minute. The radio-iodine content was reduced by 81% on passage through the filter.
- (2) When a similar volume of water containing radioactive iodide was passed through the filter the reduction in activity was approximately 98%.
- (3) On passage of 100 ml of water contaminated with extract from the air filter from Round 3 through a 10 cm x 1 cm² column of De-acidite FF (an anion exchange resin) in the chloride form, over 98% of the iodine activity was removed.
- (4) When 10 ml of the water was shaken with 1 gram of Metasil ALAG and centrifuged about 22% of the iodine activity was removed. This procedure is known to effect almost complete removal of traces of iodide from water.

APPENDIX E

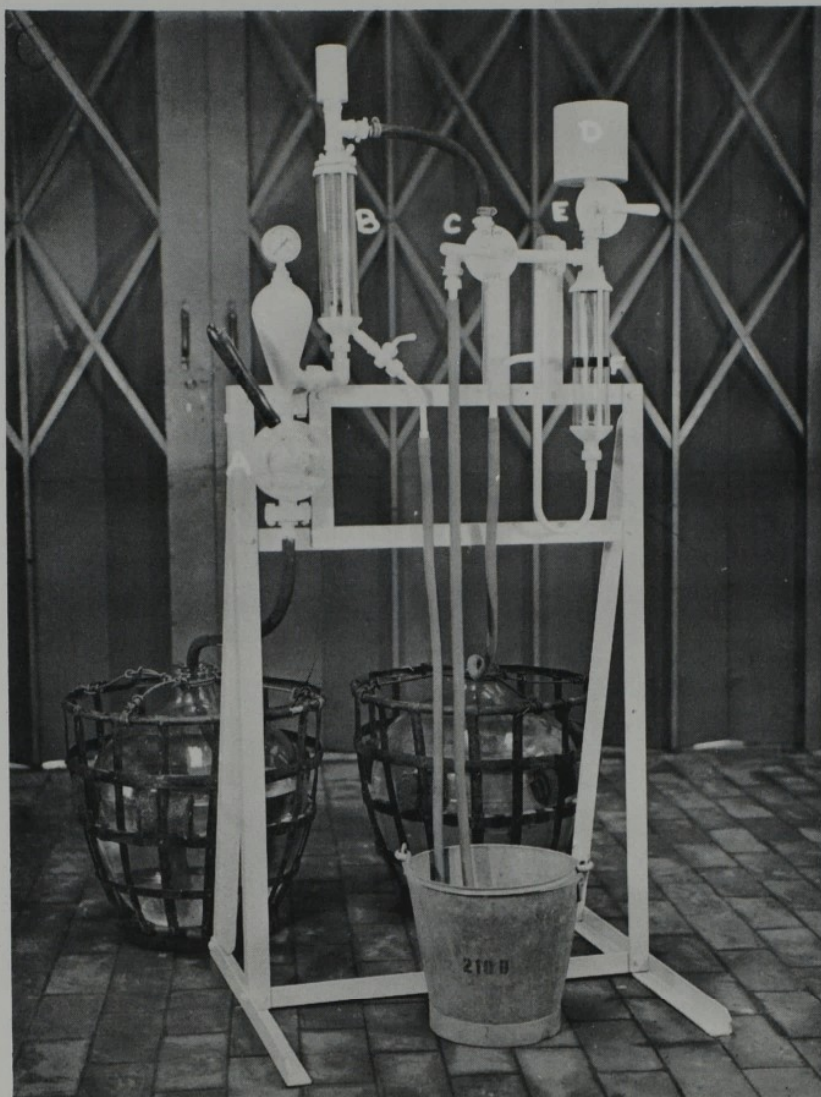
Activity to be Disposed of

Concentration of activity in final effluent acceptable for military purposes (see Section 3.1, footnote marked* on page 6) = 3×10^{-2} mc/litre.

If equipment is 95% efficient, then concentration in raw water supply can be 0.6 mc/litre.

Thus it will be possible to use water containing $0.6 \times 4.54 \times 200$ mc in 200 gallons and still produce a potable effluent. Of this, approximately 500 millicuries will be removed by the beds.

In the case of an hour's flow through a Paterson equipment, up to $0.5 \times \frac{3000}{200} = \underline{7.5 \text{ curies}}$ may be removed by the beds.



- Key: A Pump
 B Metafilter candle and filter bed
 C By-pass cock
 D Brine tank
 E Cock
 F Ion-exchange bed

The carboy on the left of the photograph contains the raw water. That on the right receives the treated effluent. Waste, washings etc., are led into the bucket.

Figure 1.

Initial Distribution

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